

10 5400 4459
Rec'd CTI PTO 22 JUN 2005

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau



PCT

(43) International Publication Date
29 July 2004 (29.07.2004)

(10) International Publication Number
WO 2004/062838 A2

(51) International Patent Classification⁷: B22F 7/00

[DE/DE]; An der Goldenen Höhe 11, 01728 Hähnichen (DE).

(21) International Application Number:
PCT/EP2003/014381

(74) Agent: PFENNING, MEINIG & PARTNER GBR;
Gostritzer Strasse 61-63, 01217 Dresden (DE).

(22) International Filing Date:
17 December 2003 (17.12.2003)

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW.

(30) Priority Data:
103 01 175.7 8 January 2003 (08.01.2003) DE

(84) Designated States (*regional*): ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

(71) Applicants (*for all designated States except US*):
INCO LIMITED [CA/CA]; 145 Street, West, Suite 1500, Toronto, Ontario M5H 4B7 (CA). FRAUNHOFER-GESELLSCHAFT ZUR FÖRDERUNG DER ANGEWANDTEN FORSCHUNG E.V. [DE/DE]; Hansastrasse 27c, 80686 München (DE).

Published:

— without international search report and to be republished upon receipt of that report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(72) Inventors; and
(75) Inventors/Applicants (*for US only*): NAUMANN, Dirk [DE/CA]; 5439 Middlebury Drive, Mississauga, Ontario LSM 5E8 (CA). WEISSGÄRBER, Thomas [DE/DE]; Am Feldrain 38, 01328 Dresden (DE). BÖHM, Alexander

WO 2004/062838 A2

(54) Title: COMPONENT PRODUCED OR PROCESSED BY POWDER METALLURGY, AND PROCESS FOR PRODUCING IT

(57) Abstract: The invention relates to components which are produced or processed by powder metallurgy, and to processes for producing components of this type. The components produced by powder metallurgy are intended both to have porous regions and to provide fluid-tight properties, and it should also be possible to produce them at correspondingly low cost and suitably flexibly. For this purpose, a component of this type has at least one porous region, which is formed from an intermetallic phase or solid solutions. However, it may also have a corresponding surface coating. Moreover, in a component of this type there is at least one areal fluid-tight region which is formed from a metal or metal alloy of the corresponding intermetallic phase or solid solution.

Component produced or processed by powder metallurgy,
and process for producing it

5 The invention relates to components which are produced by powder metallurgy or alternatively are processed by powder metallurgy and have at least one porous region, which is formed from an intermetallic phase or solid solutions, or have a surface coating of this type. In
10 addition, the invention also relates to corresponding production processes. In this context, the term processing by powder metallurgy is to be understood as meaning a corresponding, retrospective processing of semifinished products, such as for example metal foam
15 structures, by powder metallurgy.

The prior art has disclosed possible ways of producing sintered porous bodies which have been formed from intermetallic phases or solid solutions. A process of
20 this type is described, for example, in DE 101 50 948. In this document, it is proposed for a powder with a sintering activity which at least forms intermetallic phases or solid solutions to be applied to the surface of a porous base body. Then, the formation of
25 intermetallic phases or solid solutions is supposed to be initiated by means of a heat treatment. At the same time, the surface area can thereby be increased.

Although the bodies produced in this way have a
30 relatively low inherent mass and also, if suitable intermetallic phases or solid solutions are selected, a high thermal stability, they cannot readily be used for some applications. This is true in particular with regard to use as a sealing element without additional
35 assembly or connection to components which are impervious to the various fluids.

Therefore, it is an object of the invention to provide components which are produced by powder metallurgy and

have both porous regions and fluid-tight properties and which can also be produced flexibly and at low cost.

According to the invention, this object is achieved by 5 components which have the features of claim 1. Advantageous production processes result in accordance with claims 10, 13 and 14. Advantageous configurations and refinements of the invention can be achieved by the features listed in the subclaims.

10 The component according to the invention which is produced by powder metallurgy or is additionally processed in this way accordingly includes at least one porous region, which is formed from an intermetallic phase or solid solutions. However, a porous region of 15 this type may also be provided with a corresponding surface coating which is formed from an intermetallic phase or solid solutions of this type.

20 Furthermore, there is at least one areal fluid-tight region which is formed from a metal, a metal alloy of the corresponding intermetallic phase or the corresponding solid solution.

25 The term fluid-tight is to be understood as meaning at least imperviousness to certain liquids, but also, under certain circumstances, gas-tightness and even imperviousness to low-molecular gases or gases with a low atomic number.

30 In an advantageous configuration, the fluid-tight region may form part of the outer shell of the component, which the correspondingly porous region may then adjoin in one direction.

35 However, it is also possible for a fluid-tight region of this type to be surrounded by the porous region. In this case, the fluid-tight region may form a type of core or alternatively a barrier within a component.

Nickel, aluminum, molybdenum, tungsten, iron, titanium, cobalt, copper, silicon, cerium, tantalum, niobium, tin, zinc or bismuth can be used to form the intermetallic phases or solid solutions. It has proven particularly advantageous for at least the porous region to be made from nickel aluminide or to use a corresponding surface coating made from nickel aluminide, since this also makes it possible to achieve very good thermal stabilities.

However, the porous region may advantageously also be formed in such a way that a porosity changes in the direction of the areal, fluid-tight region. This may be effected in steps, i.e. in layers with different porosities within the individual layers, or a continuously graduated form.

The fluid-tight region should advantageously have a density which is over 96% of the corresponding theoretical density.

In one embodiment, however, the fluid-tight region may be formed from a pure metal or a metal alloy of the corresponding intermetallic phases or of a solid solution which is formed areally, for example in the form of a plate. For example, a porous region can be arranged on a nickel component which is, for example, of plate-like design and a porous region, which either consists of nickel aluminide or is surface-coated with nickel aluminide, can be joined by material-to-material bonding to it, as described in more detail below.

Furthermore, it is possible for at least one passage or an aperture to be formed within the fluid-tight region. A passage can be used, for example, for liquid or gaseous coolant to pass through. However, it is also possible to use a passage of this type and adjoining openings to generate a reduced pressure all the way

into the porous region, so that a sucking or vacuum action can be achieved in that region.

However, apertures can also be used to secure a component according to the invention using mechanical means.

There are a number of alternative options for producing and/or coating components according to the invention.

For example, to produce components of this type, it may be expedient to use different starting powders. In this case, a starting powder which has a sintering activity and forms intermetallic phases or solid solutions should be used at least to form an areal, fluid-tight region. This makes it possible to make use of the effect whereby an increase in volume is observed during sintering, causing sufficiently dense sintering of the corresponding region, so that the required fluid-tightness can be achieved.

Starting powders with a mean grain size $d_{50} < 50 \mu\text{m}$ should be used in particular to form the porous region during sintering, it being possible, for example, to form the stepped or graduated porous regions which have already been mentioned above to be formed by means of a suitable selection of different grain size fractions.

However, it is also possible, in order to produce components according to the invention, to produce starting powders of the abovementioned grain size fraction in combination with a powder which has a sintering activity and is obtained by high-energy milling.

For example, a porous region may be formed exclusively from a starting powder of this type, while an adjoining region, which is likewise porous, may be formed by means of a mixture of this starting powder with a

powder which has a sintering activity and is obtained by high-energy milling, and for a fluid-tight region then to be formed exclusively by means of a starting powder which has a sintering activity and is obtained
5 by high-energy milling.

These different powders employed have different properties during the sintering. In this context, in particular the differing shrinkage is of importance.

10 For example, a powder preform which has been prepared for the powder metallurgy production of components according to the invention may have locally differing dimensions which take account of the different starting
15 powders and their shrinkages which are observed during sintering, so that after sintering a component which is at least near net shape can be provided, requiring at most only slight remachining.

20 During production of a powder preform of this type, by way of example regions in which the powder preform contains starting powders with a higher sintering activity, such as for example powder mixtures obtained by high-energy milling, or have been formed in such
25 regions exclusively from powders of this type with corresponding binders, are characterized by higher shrinkages, which have to be taken into account accordingly.

30 In another alternative, however, it is also possible for components according to the invention to be produced in such a way that a porous structure which is to form the porous region has already been areally coated with a powder which has a sintering activity and
35 forms intermetallic phases or solid solutions. Then, the coated region can be formed in a fluid-tight manner on the corresponding surface of the components by means of a sintering operation.

In this case, by way of example, it is possible to use a porous starting structure such as a semifinished product, comprising a corresponding intermetallic phase or a solid solution.

5

However, it is also possible for a porous structure, likewise in the form of a semifinished product, such as a metal foam, preferably a nickel foam, to be surface-coated with a powder which forms intermetallic phases or solid solutions, as is known from DE 101 50 948, and for an areal layer then additionally to be formed on a surface from a powder which has a sintering activity and forms intermetallic phases or solid solutions and which then likewise forms the fluid-tight region during sintering. For example, the porous structure, i.e. the porous region of a component according to the invention, can be correspondingly modified and the fluid-tight region formed in a sintering operation.

20 A further alternative production option consists in a metallic element, which is areal and fluid-tight at least in regions and is to form the fluid-tight region, to be joined to a porous structure, which then forms the porous region, by material-to-material bonding.

25 This can be achieved by means of a sintering operation in which the metallic areal element is coated beforehand with a layer of a powder which contains at least one element of the intermetallic phase or of the corresponding solid solution and forms a material-to-material bond with this powder during sintering. The metallic areal element may likewise be formed from an element of the corresponding intermetallic phase or solid solution or from an alloy of this element.

30 35 The invention is to be described below by way of example.

Example 1

5 A starting powder mixture which contains nickel and aluminum was used to produce an example of a component according to the invention. The grain size fraction was in the range between 5 and 30 µm.

A nickel to aluminum atomic ratio of 50/50 atomic % was maintained for the mixture composition. The nickel and 10 aluminum starting powders were mixed with one another for a period of 0.5 h. This mixture M1 was then divided into two partial quantities. One of these partial quantities was subjected to high-energy milling in a Fritsch P5 planetary ball mill at a rotational speed of 15 250 min/h for a period of 1 h. This resulted in a part mixture M2. In turn, a third part mixture M3 was produced from the mixture M1 and the mixture M2, containing these two mixtures in equal parts.

20 Components were compacted from these mixtures in advance by die-pressing in the following order: mixture M1, mixture M2 and mixture M3.

Then, a reaction sintering operation was carried out in 25 vacuo at a temperature in the region of 1150°C, and a component according to the invention which has three different porous regions was produced. That part of the component which was formed from powder mixture M3 forms the fluid-tight region, whereas the regions formed from 30 mixtures M1 and M2 had a significantly higher porosity.

It was possible to use the powder mixtures with conventional binders which are known per se and are removed during sintering. The grain sizes of the 35 different starting powders M1 to M3 were kept virtually constant, and accordingly in this example there is no grain size change in the high-energy milling process, only the sintering activity of the powder having been changed.

Example 2

A nickel foam structure is surface-coated with a pure
5 aluminum powder or a nickel-aluminum powder obtained by
high-energy milling. A nickel/aluminum atomic ratio in
the range between 75 to 50 atomic % of nickel to 25 to
50 atomic % of aluminum was maintained. The coating
with a powder of this type was carried out in such a
10 way that an open porosity of the nickel foam was
retained. The nickel foam body prepared in this way was
then coated on one side with a powder M3 as described
in Example 1, after which sintering was again carried
out at a temperature of approx. 1150°C. The
15 corresponding intermetallic phases were formed on the
surface of the nickel foam, and a fluid-type region
comprising nickel aluminide was formed where the powder
M3 was additionally applied.

Patent Claims

1. A component which is produced or processed by
5 powder metallurgy and has at least one porous region,
which is formed from an intermetallic phase or solid
solutions or has a surface coating of this type, and at
least one areal fluid-tight region, which is formed
from a metal, a metal alloy, the corresponding
10 intermetallic phase or solid solution.

2. The component as claimed in claim 1, wherein the
fluid-tight region forms part of the outer shell of the
component.

15 3. The component as claimed in claim 1, wherein the
areal fluid-tight region is surrounded by the porous
region.

20 4. The component as claimed in one of the preceding
claims, wherein the corresponding intermetallic phase
or the solid solutions are based on nickel, aluminum,
molybdenum, tungsten, iron, titanium, cobalt, copper,
silicon, cerium, tantalum, niobium, tin, zinc or
25 bismuth.

5. The component as claimed in one of the preceding
claims, wherein at least the porous region is formed
from nickel aluminide or is coated therewith.

30 6. The component as claimed in one of the preceding
claims, wherein at least the porous region has a
porosity and density which change in steps or gradually
in the direction of the areal fluid-tight region.

35 7. The component as claimed in one of the preceding
claims, wherein the areal fluid-tight region is formed
from a metal or metal alloy of the corresponding
intermetallic phase or solid solution.

8. The component as claimed in one of the preceding claims, wherein at least one passage or aperture is formed in the areal fluid-tight region.

5

9. The component as claimed in one of the preceding claims, wherein the areal, fluid-tight region has a density of over 96% of the theoretical density.

10 10. A process for producing the component as claimed in claim 1 by powder metallurgy, wherein a starting powder which has a sintering activity and forms intermetallic phases or solid solutions is used to form the areal fluid-tight region.

15

11. The process as claimed in claim 10, wherein a starting powder with a grain size $d_{50} < 50\mu\text{m}$ and a powder with a sintering activity obtained by high-energy milling are used for production.

20

12. The process as claimed in claim 11, wherein a powder preform is produced from differentiated starting powders, the dimensions of which preform take account of the different shrinkages of the differentiated starting powders during sintering.

25 13. A process for producing the component as claimed in claim 1, wherein a porous structure, which forms the porous region, is coated with a powder which has a sintering activity and forms intermetallic phases or solid solutions, and the areal fluid-tight region is formed at a surface of the component by a subsequent sintering operation.

30 35 14. A process for producing the component as claimed in claim 1, wherein a metallic, areal and fluid-tight element, which forms the fluid-tight region, is coated with a layer of a powder which contains at least one element of the intermetallic phase or solid solution,

and the fluid-tight region is joined to a porous structure, which has been placed on top of the powder layer and forms the porous region, by sintering.

10/54 0459
Rec'd PCT/PTO 22 JUN 2005

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization International Bureau



(43) International Publication Date
29 July 2004 (29.07.2004)

PCT

(10) International Publication Number
WO 2004/062838 A3

(51) International Patent Classification⁷: B22F 7/00, (74) Agent: PFENNING, MEINIG & PARTNER GBR;
3/11, 7/06 Gostritzer Strasse 61-63, 01217 Dresden (DE).

(21) International Application Number:
PCT/EP2003/014381

(81) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW.

(22) International Filing Date:
17 December 2003 (17.12.2003)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
103 01 175.7 8 January 2003 (08.01.2003) DE

(71) Applicants (for all designated States except US):
INCO LIMITED [CA/CA]; 145 Street, West, Suite 1500, Toronto, Ontario M5H 4B7 (CA). FRAUNHOFER-GESELLSCHAFT ZUR FÖRDERUNG DER ANGEWANDTEN FORSCHUNG E.V. [DE/DE]; Hansastrasse 27c, 80686 München (DE).

(72) Inventors; and
(75) Inventors/Applicants (for US only): NAUMANN, Dirk [DE/CA]; 5439 Middlebury Drive, Mississauga, Ontario L5M 5E8 (CA). WEISSGÄRBER, Thomas [DE/DE]; Am Feldrain 38, 01328 Dresden (DE). BÖHM, Alexander [DE/DE]; An der Goldenen Höhe 11, 01728 Hähnichen (DE).

(84) Designated States (regional): ARIPO patent (BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

- with international search report
- before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments

(88) Date of publication of the international search report:
29 December 2004

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 2004/062838 A3

(54) Title: POWDER METALLURGICAL PRODUCTION OF A COMPONENT HAVING POROUS AND NON POROUS PARTS

(57) Abstract: The invention relates to components which are produced or processed by powder metallurgy, and to processes for producing components of this type. The components produced by powder metallurgy are intended both to have porous regions and to provide fluid-tight properties, and it should also be possible to produce them at correspondingly low cost and suitably flexibly. For this purpose, a component of this type has at least one porous region, which is formed from an intermetallic phase or solid solutions. However, it may also have a corresponding surface coating. Moreover, in a component of this type there is at least one areal fluid-tight region which is formed from a metal or metal alloy of the corresponding intermetallic phase or solid solution.

A. CLASSIFICATION OF SUBJECT MATTER
 IPC 7 B22F7/00 B22F3/11 B22F7/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
 IPC 7 B22F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 01/70436 A (YAHIA L HOCINE ; VICTHOM CORP SOC (CA); JOUIN JACQUES (FR); DAUMAS MAR) 27 September 2001 (2001-09-27) claim 8; example 2	1,3-5,7, 9
X	US 5 634 189 A (ROSSMANN AXEL ET AL) 27 May 1997 (1997-05-27) claims 8,9	1,2,6
X	US 6 241 469 B1 (NAZMY MOHAMED ET AL) 5 June 2001 (2001-06-05) claims 1,2,9	1,8
X	EP 0 657 950 A (KATAYAMA TOKUSHU KOGYO KK) 14 June 1995 (1995-06-14) column 11, line 13 - line 44; claim 2	1,10,11
	-/-	

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

° Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the International filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the International filing date but later than the priority date claimed

- "T" later document published after the International filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

8 October 2004

Date of mailing of the International search report

20/10/2004

Name and mailing address of the ISA
 European Patent Office, P.B. 5818 Patentlaan 2
 NL - 2280 HV Rijswijk
 Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
 Fax (-31-70) 340-3016

Authorized officer

Schruers, H

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2002/172836 A1 (TAKAYAMA TAKEMORI ET AL) 21 November 2002 (2002-11-21) paragraphs '0037!, '0041!, '0042!, '0049!; claim 13 -----	1,14
X	US 4 855 101 A (MOHS RUDOLF ET AL) 8 August 1989 (1989-08-08) column 2, line 12 - line 24; claims 1,12 -----	1,14
A	WO 01/21347 A (ROCKWELL SCIENCE CT LLC) 29 March 2001 (2001-03-29) page 6, column 34 - page 7, column 15; claim 1 -----	12

Patent document cited in search report		Publication date		Patent family member(s)		Publication date
WO 0170436	A	27-09-2001	FR AU WO	2806421 A1 4427701 A 0170436 A1		21-09-2001 03-10-2001 27-09-2001
US 5634189	A	27-05-1997	DE FR GB	4338457 A1 2712218 A1 2284825 A ,B		18-05-1995 19-05-1995 21-06-1995
US 6241469	B1	05-06-2001	DE DE EP	19848104 A1 59907926 D1 0995880 A2		20-04-2000 15-01-2004 26-04-2000
EP 0657950	A	14-06-1995	JP JP CA CN DE DE EP US US	3509031 B2 7166211 A 2136804 A1 1109200 A ,B 69429153 D1 69429153 T2 0657950 A1 5508114 A 5655295 A		22-03-2004 27-06-1995 11-06-1995 27-09-1995 03-01-2002 14-11-2002 14-06-1995 16-04-1996 12-08-1997
US 2002172836	A1	21-11-2002	JP US	2002180216 A 2004123698 A1		26-06-2002 01-07-2004
US 4855101	A	08-08-1989	DE EP	3723650 A1 0299238 A2		26-01-1989 18-01-1989
WO 0121347	A	29-03-2001	US CA EP JP WO	6517773 B1 2382369 A1 1216115 A1 2004514051 T 0121347 A1		11-02-2003 29-03-2001 26-06-2002 13-05-2004 29-03-2001